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# Adequacy of Surface Analytical Tools for Studying the Tribology of Ceramics

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# ADEQUACY OF SURFACE ANALYTICAL TOOLS FOR STUDYING

## THE TRIBOLOGY OF CERAMICS

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### SUMMARY

Surface analytical tools are very beneficial in tribological studies of ceramics. Traditional methods of optical microscopy, XRD, XRF, and SEM should be combined with newer surface sensitive techniques especially AES and XPS. ISS and SIMS can also be useful in providing additional composition details. Tunneling microscopy and electron energy loss spectroscopy are less known techniques that may also prove useful.

### INTRODUCTION

Currently, there are many surface analytical tools available. There are an estimated 70 to 100 different techniques that can be used to examine surfaces and surficial layers. These techniques provide various types of information on surface microtopography, crystallography, or chemical composition. There is considerable overlap in the information that can be derived from these techniques; also, some differ from each other in relatively minor aspects of their methodology and information output. Relatively few of the known techniques, those which the writer considers of significant usefulness to the tribologist, will be discussed.

Some of the older methods of examining surfaces and surficial regions should not be ignored in the rush to employ the most recent surface analytical techniques. This is especially true in the tribological studies of ceramics. In fact, ceramics, because most of them are electrical insulators, pose certain problems to the analyst employing some of the very surface sensitive techniques of electron and ion spectroscopy.

The obvious, traditional techniques that can be very useful in the study of ceramics are optical microscopy, x-ray diffraction (XRD), x-ray fluorescence (XRF), and stylus profilometry. XRD and XRF are not really surface sensitive because the spectral information derived is from a surficial region whose depth depends on the energy of the analyzing x-ray beam and absorbance of the specimens, but typically is on the order of 0.02 mm in depth. These methods obviously are not for monolayer analysis, but the surficial region probed is also a region of great interest to the tribologist because, for example: Hertzian stresses, load and friction induced plastic deformation, recrystallization layers, residual stresses, and products of surface-initiated chemical reactions all are frequently present in this region. All of the traditional techniques mentioned can be used in an air atmosphere while the electron and ion beam techniques usually require a vacuum environment.

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Notwithstanding the problems associated with electron and ion beam techniques in the study of ceramics, some of these techniques can provide extremely valuable information. Rapid progress is also being made in getting around some of the inherent problems such as electrical charge build-up on specimens of insulator materials.

The analytical methods to be discussed are classified in this paper as: (1) traditional methods such as x-ray diffraction, optical microscopy, and stylus profilometry; (2) well-established electron beam analysis techniques such as scanning electron microscopy (SEM), low energy electron diffraction (LEED), Auger electron spectroscopy (AES), and x-ray photoelectron spectroscopy (XPS); (3) ion beam analysis techniques such as ion scattering spectroscopy (ISS), and secondary ion mass spectroscopy (SIMS); and (4) electron energy loss spectroscopy (EELS), and tunneling microscopy.

Where it appears useful, a brief apparatus description will be given. Examples of application to tribological studies will be emphasized.

## INSTRUMENTS AND APPLICATIONS

### Traditional Methods

The importance of the type of information that can be obtained by x-ray diffraction (XRD) is well known. This information, which includes crystallography, grain size, stress distribution, and glass-phase detection are all of obvious importance in the tribological behavior of ceramics and do not require elaboration here.

Optical microscopy is also of obvious value, but some of the techniques of great value in studying mechanisms of both liquid and solid lubrication should be mentioned. The information explosion in the 1960's and 1970's concerning the mechanisms of elastohydrodynamic lubrication with oils was to a significant extent due to the use of optical interferometric microscopy. With this method, it is possible to determine the lubricant film thickness distribution, and pressure distribution within a lubricated contact. Combined with infrared microscopy (as pioneered by Winer et al. at the Georgia Institute of Technology) temperature distribution within a lubricated, concentrated contact can also be determined. Figure 1 is a schematic of an optical metallograph combined with a tribometer for the study of Hertzian contacts. Optical tribometers were originally developed to study oil films in lubricated contacts (ref 1.). When rolling or sliding is initiated, an oil film is formed in the contact and the color distribution (due to optical interferometric effects) can be used to compute film thickness distribution.

The behavior of solid lubricants within concentrated contacts can also be studied with this apparatus (ref. 2). Figure 2 for example, shows the passage of a solid lubricant through a sliding concentrated (ball on flat) contact with an average contact pressure of 1 GPa (150 000 psi). A qualitative model of solid lubricant behavior deduced from such photographs incorporates the following steps: (1) the lubricant particles enter the contact, (2) then coalesce into a thin film between the sliding surfaces, and (3) solid lubricants function by a shear mechanism characterized by extreme continuous plastic deformation and flow.

Figures 3 and 4 provide a contrast to the behavior of a solid lubricant with the behavior of abrasive particles and of unlubricated glass. Abrasive particles also enter the contact, but instead of deforming plastically, they fracture, imbed in the softer sliding surfaces and cause extreme abrasion. The unlubricated glass is interesting in that the crack initiation and propagation are clearly seen to occur as could be predicted by the calculated surface stress pattern shown below the photomicrographs.

Stylus profilometry is also of obvious value, but is sometimes damaging to the film because it is a contact technique. Several noncontact techniques are now available. One of these is tunneling microscopy which will be discussed in a later section.

### Electron Beam Analysis

Scanning electron microscopy (SEM) is a basic tool for the tribologist. Combined with energy dispersive spectroscopy (EDS) or (EDAX), it can give valuable information on surface morphology and composition. Figure 5 shows examples of an SEM and x-ray dot maps which give the element distribution in a metal/nonmetal composite (ref. 3). Ceramics tend to "charge-up" in the SEM and therefore are usually coated with an extremely thin metallic or carbon film. The EDS technique probes to a sufficient depth to obtain elemental analysis of the ceramic substrate.

Low energy electron diffraction (LEED) is a well established surface sensitive technique. Because the incident beam is low energy (40 to 400 eV), structural (crystallographic) information can be obtained for monolayer surface films. Figure 6 is a schematic of a LEED instrument combined with an adhesion experiment. Figure 7 is an example of LEED patterns for a clean iron surface compared with an iron surface with an absorbed monolayer of methane (ref. 4). Unfortunately, LEED can only be used with difficulty or not at all for insulators such as ceramics because they readily assume an electrostatic charge in an electron beam; since the incident beam is of very low energy, conductive coatings do not appear to be a solution. However, LEED is applicable to conductive ceramics such as silicon carbide and semi-conductors solid lubricants such as molybdenum disulfide. Another limitation of LEED is that its use is limited for the most part to the study of single crystals.

Auger electron spectroscopy (AES). - AES is also a surface sensitive technique. It uses an electron beam typically of 1 to 5 keV energy and probes slightly deeper than LEED. AES gives composition rather than structural information. Probe depths can be several monolayers below the surface, but the technique is sensitive to a fraction of a monolayer. Auger analysis yields elemental composition and some information on the chemical bond state of those elements. The use of Auger to analyze insulators presents experimental difficulties because of electrostatic charging, however procedures have been developed for obtaining spectra of insulators including glass (ref. 5). In that reference, excellent examples are given of glass spectra before and after sputter etching which show significant differences in the surface composition compared to the near-surface (surficial) layer composition.

Figure 8 is a schematic of a combined Auger system and a pin on disk tribometer. This combination allows surface analysis of a spot on the disk

wear track immediately after it exits the sliding contact, thus minimizing changes in surface composition that could occur in transferring wear specimens from a tribometer to a separate Auger system. An incidental benefit of analyzing a moving surface, which is applicable as well to nontribological studies is that problems of electrostatic charging are reduced by presenting an ever changing target area to the incident electron beam.

X-ray photoelectron spectroscopy (XPS or ESCA) differs from AES in that it employs monoenergetic (usually soft) x-rays to excite emission of characteristic photoelectrons from the specimen surface. The energies of the emitted electron are characteristic of the elements present and their chemical bond states. Compared to Auger, XPS gives more complete and more easily interpreted information on chemical bond states. For example sulfur can be identified as elemental sulfur, sulfide, sulphate, etc. XPS can also be combined with a tribometer in a vacuum system.

Figure 9 gives the XPS spectra for silicon carbide (SiC) in vacuum at various temperatures (ref. 6). The ability of XPS to detect bond states enables the analyst to differentiate carbon as amorphous carbon, graphite or carbide from the binding energies of the carbon peaks in the spectra. In this example, it is clear that the initially-present amorphous carbon disappears with increasing temperature and graphite appears at the surface. This has been interpreted to mean that silicon carbide dissociates at high temperature to graphite and to elemental silicon which sublimates away. The results of friction experiments in vacuum (fig. 10) are in agreement with this interpretation. As initial surface films are desorbed with increasing temperature, the friction coefficient increases dramatically. However, as graphite appears on the surface the friction coefficient decreases to its room temperature value. Figure 11 illustrates that different results can be expected in air, where silicon carbide shows a decrease in friction above about 400 °C. At high temperatures in air the surface becomes covered with a film of silica. The metallic counterface material also oxidizes which contributes to reduced friction. Any graphite formed is oxidized to carbon monoxide or dioxide above 500 °C.

### Ion Beam Analysis

Ion scattering spectroscopy (ISS) and secondary ion scattering mass spectroscopy (SIMS) utilize analyses by mass spectrometry in contrast to the previously discussed techniques that utilize electron spectroscopy. An ion spectroscopy schematic is shown in figure 12.

In principle, ISS is extremely simple. A beam of ions is aimed at a surface at an oblique angle and the scattered ions are energy analyzed to give the mass of surface ions from which they elastically scattered. The technique is surface sensitive and gives elemental and quantitative information.

Ion scattering can be used to some extent as a channeling technique to probe very thin films in depth. Ions scattered from the surface will have a slightly different energy than those scattered from the film/substrate interface (ref. 7). The energy peaks will not be totally resolved in the spectra but can be mathematically resolved into their component peaks.

SIMS is very surface sensitive and is capable of analyzing organic molecular groupings on a surface. Research results reported in (ref. 8) for example, demonstrate the usefulness of this tool for adsorption/desorption studies of organics on bearing materials.

Electron energy loss spectrometry (EELS) is a high resolution surface sensitive analytical tool which provides information about the vibrational motion of surface atoms and molecules under ultra high vacuum conditions. The technique literally generates an infrared spectra of the surface. Figure 13 is a schematic diagram of an EELS system. A monoenergetic beam of electrons is impinged upon the sample surface where they interact with surface atoms and molecules to excite their characteristic vibrational motion. In so doing, the incident electrons lose energy in a manner quantitatively related to the characteristic vibrations of the surface species. The exciting beam therefore contains information about the surface vibrational states.

An elegant example of an EELS study was reported by J. Wacławski (ref. 9). The energy loss spectra of diamond before and after heating to 1000 °C in UHV were obtained. The "as-polished" diamond contains peaks for vibrational modes characteristic of the hydrogen to carbon bond, literally hydrocarbon (HC) bonds. After heating and hydrogen desorption the HC peaks are no longer present. This correlates with studies reported by Pepper (ref. 10) concerning the effect of temperature on the friction of copper sliding on diamond, figure 14. Below about 800 °C friction was low, but rose sharply at higher temperatures. Pepper was able to relate this phenomenon both theoretically and experimentally to changes in the electron surface state of the diamond caused by thermal desorption of hydrogen.

EELS is most applicable to surface studies of single crystal conductors and semi-conductors. However, the use of a secondary electron gun to control charging effects enables one to also study insulating materials (ref. 11).

#### HYDROGEN DETECTION

EELS may be an especially valuable analytical tool for ceramics because of its ability to detect hydrogen. The tribology of ceramics is sensitive to the presence of hydrogen and its state of chemical combination on the surface. Other techniques with some potential for distinguishing between atomic hydrogen, hydroxyl, water, and hydrocarbon species include the following:

- (1) secondary ion mass spectroscopy (SIMS) with a reliable means of compensating for charge effects
- (2) emission infrared spectroscopy
- (3) Raman spectroscopy

The optical techniques have an advantage in allowing measurements to be made in air; vacuum is not required.

#### Tunneling Microscopy

The tunneling microscope is a noncontact method of surface profilometry. An electron emitter with a typical radius of 100 to 10 000 Å is brought close to a conducting surface while a contact current is passed through the

emitter. The field strength is related to the emitter to surface spacing. The emitter is swept across the surface by two piezoelectric elements. The emitter is mounted on a third piezoelectric element which moves the emitter in the z-axis during the sweep. The z-axis movement maintains a constant spacing via a feedback circuit that maintains constant current. As the emitter rasters the surface, an xy topographic representation of the surface is recorded.

In 1972 (ref. 12), resolution was adequate to provide topographic maps of a diffraction grating with 180 lines per mm spacing. Currently resolution is sufficient to depict individual atoms and their arrangements on a surface (ref. 13). Again, this method will require special techniques, if applicable at all, to be used on insulating ceramics.

### COMPARISONS OF CHARACTERIZATION TECHNIQUES

Traditional techniques such as optical microscopy and x-ray diffraction can be used in an air atmosphere while the electron and ion beam techniques usually require a vacuum environment. Charged particle beams produce electrostatic charges on the surface of insulating materials such as most ceramics.

Notwithstanding the problems associated with electron and ion beam techniques in the study of ceramics, some of these techniques can provide extremely valuable information. Rapid progress is being made in getting around some of the inherent problems such as electrical charge build-up on insulators.

The most common indispensable electron beam method is scanning electron microscopy (SEM). The problem of charge build-up is easily solved by coating insulator specimens with a thin film of a conducting material such as graphite or vapor deposited gold. SEM has the advantages of excellent depth of focus (rough surfaces may be examined) and very high magnification capability. SEM requires that the specimen have some composition or topographical variation on the specimen surface in order to produce a sharp image of sufficient contrast. Therefore, a very smooth surface of uniform composition will produce a featureless image on SEM. SEM also requires a vacuum atmosphere in the specimen chamber. Optical microscopy should therefore be used as a complimentary tool to SEM. Often surfaces that are featureless on SEM show very good contrast on an optical microscope. Conversely, the optical microscope requires a very smooth surface and is usually unsatisfactory except at low magnification on the relatively rough surfaces characteristic of wear areas.

Of the truly surface sensitive analytical tools available, some of the most useful to the tribologist are Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), ion scatter spectroscopy (ISS), and secondary ion mass spectroscopy (SIMS).

AES is capable of identifying all elements except hydrogen and helium. It can detect surface species present in a concentration of as little as 1/100 of a monolayer. It can provide limited information on the state of combination of surface elements. For example, it can differentiate carbon present as CO, amorphous carbon, or graphite.

XPS is more powerful than AES in providing information on molecular composition (compound identification). This technique employs a monochromatic x-ray beam for excitation of surface atoms to emit photoelectrons of characteristic energies. These characteristic energies are related to the binding energies of the surface atoms from which in turn, the nature of surface molecules can be deduced. Calibrations can be done from theoretical considerations or directly from spectra of known compounds. In comparing AES and XPS, it can be said in general that AES has better surface spatial resolutions (much smaller target spot size) and can provide superior fine detail, while XPS averages over a large area but provides more molecular information. Both methods can be coupled with sputtering to provide depth profiling capability.

ISS is one of the most surface sensitive techniques, has relatively low resolution especially for high ion mass numbers and results can be difficult to interpret. However, it is of interest because of its extreme surface sensitivity. SIMS differs in detail from ISS in that while scattering of a primary beam by a surface is analyzed in ISS, the emission of secondary ions produced by a primary ion beam are analyzed in SIMS.

One of the primary limitations of the truly surface sensitive methods is related to that very sensitivity. Namely the analyses must be performed in vacuum. (The presence of an air path would interfere with the emitted electrons or ions before they could reach the detectors.) However "post facto" studies of wear specimens run in an air or other "dirty" atmosphere can yield much useful information. Of course the analyst must consider the possibility that the surface analyzed may not be exactly the same composition as it was "in situ." Dynamic analysis "in situ" during an experiment is always desirable but not always practical.

The electrical charging of insulators such as most ceramics during analyses by electron or ion beam techniques can sometimes be circumvented by adjusting primary beam energies and angles of incidence to give a stable charge on the surface. This charge can then be "stripped out" as background. It is a varying charge that is difficult to handle. In the case of ion beam techniques, the surface charge can be positive and therefore neutralized with a carefully controlled auxiliary electron beam.



A comparison of the capabilities of many surface analytical techniques is given below:

#### WIDELY USED TECHNIQUE FOR SURFACE ANALYSIS

	Surface Sensitive	Commercially Available	Analysis of Practical Systems	Elemental Analysis	Compound	Quantitative
1. Elemental and chemical results by electron levels						
AES	YES	YES	YES	YES	To a degree	To a degree
ESCA-XPS	YES	YES	YES	YES	YES	To a degree
UPS	YES	YES	NO	NO	NO	NO
APS	YES	NO	YES	YES	NO	To a degree
EM	NO	YES	YES	YES	NO	YES
XF	NO	YES	YES	YES	NO	YES
2. Chemical and Elemental analysis by mass						
ISS	YES	YES	YES	YES	NO	To a degree
RBS	NO	YES	YES	YES	NO	YES
SIMS	YES	YES	YES	YES	To a degree	To a degree
IMP	NO	YES	YES	YES	NO	YES
3. Elemental analysis by vibrational state						
ELS	YES	YES	NO	NO	NO	NO
4. Structural analysis, macroscopic features						
SEM	YES	YES	YES	YES with EDX	NO	NO
ELL	YES	YES	YES	NO	NO	NO
5. Structural analysis microscopic						
EXAFS	YES	NO	YES	NO	NO	NO
LEED	YES	YES	NO	NO	NO	NO
FIM	YES	YES	NO	YES with an Atom probe	To a degree	NO

#### CONCLUDING REMARKS AND FUTURE TRENDS

The capabilities among existing surface analytical tools enable the tribologist to study some of the chemical and physical phenomena that occur during the friction and wear process. Much can be learned about tribochemical

effects that are of primary importance to the friction and wear processes. The best experimental approach is to perform the analyses during the friction process. A limitation with the sensitive electron and ion beam techniques is that dynamic studies of this sort can only be performed in a vacuum. Post experiment analyses can be useful but can also be misleading if proper consideration is not given to the possibility of changes in surface composition when the specimen is transposed from the friction apparatus to the analytical instrument. Therefore there is a trend toward unified designs whereby the specimens can be rapidly transferred from the tribometer to the analytical chamber without removing them from the apparatus. This of course does not eliminate the problem, but it certainly minimizes it.

The problem of electrostatic charging of nonconducting specimens subjected to electron and ion beam analytical probes was discussed extensively within the text. There is currently a strong trend among surface scientists to find ways around this problem. Considerable progress has been made, and analyses that were considered impossible as little as a year ago can now be performed. More research is necessary to put the analyses of ceramics by charged beam techniques on a routine basis.

A serious limitation is the difficulty in using beam techniques to reveal the presence of hydrogen and hydroxyl or water molecules on the surface. Electron energy loss spectroscopy has been mentioned as one technique for hydrogen detection. Other possibilities are optical techniques such as emission infrared spectroscopy and Raman spectroscopy.

Anticipated future trends include the increasing use of electron energy loss spectroscopy and possibly ion tunneling techniques. An interesting pioneering study is being conducted at NASA Lewis Research Center on possible uses of acoustic microscopy in tribological studies.

Useful analytical techniques are in place. They are constantly being improved but new ones are needed especially for the analysis of hydrogen and hydrogen radicals.

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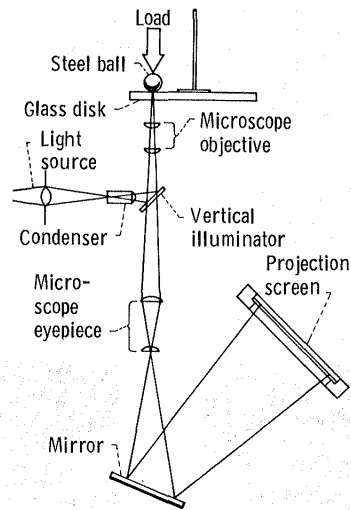
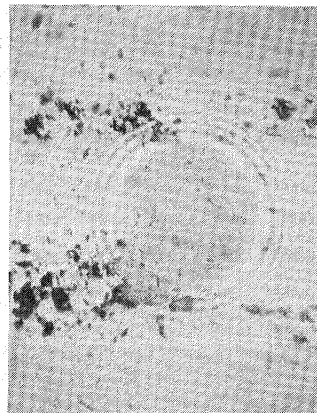
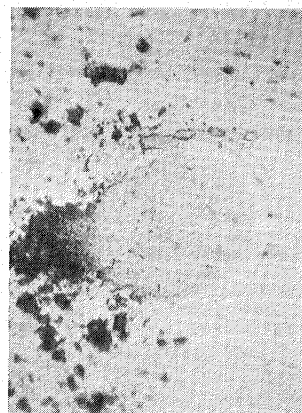


Figure 1. - Schematic of optical system.



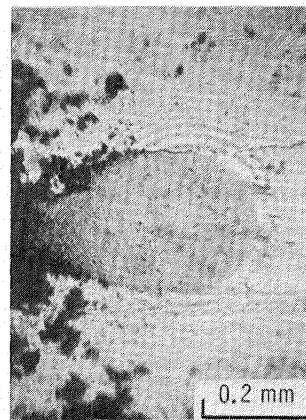
FIRST PARTICLES ENTERING  
CONTACT



PROGRESSIVE FILM  
FORMATION



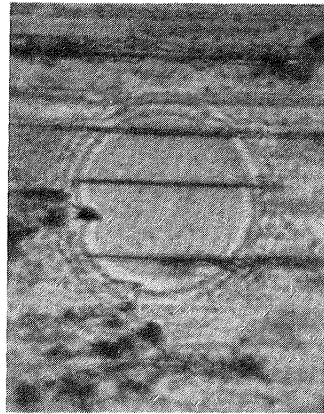
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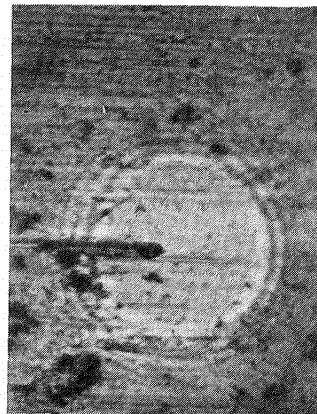
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AFTER ONLY 10 mm OF SLIDING

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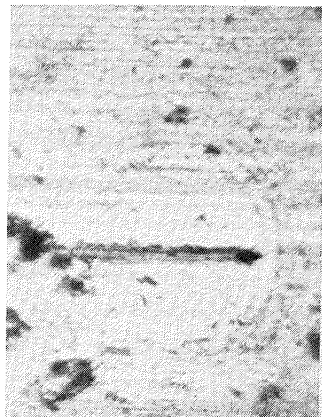
Figure 2. - Lubricating behavior of graphite fluoride.



SIC PARTICLE ENTERING  
CONTACT



PARTICLE PART WAY  
THROUGH CONTACT



PARTICLE AT CONTACT EXIT

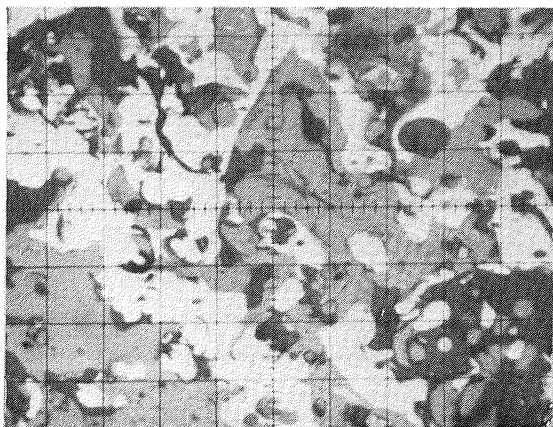


CONTACT AFTER 10 DISK  
REVOLUTIONS

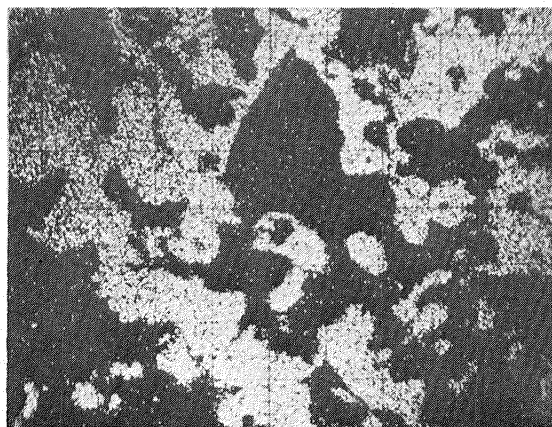
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Figure 3. - Abrasive action of silicon carbide particles.

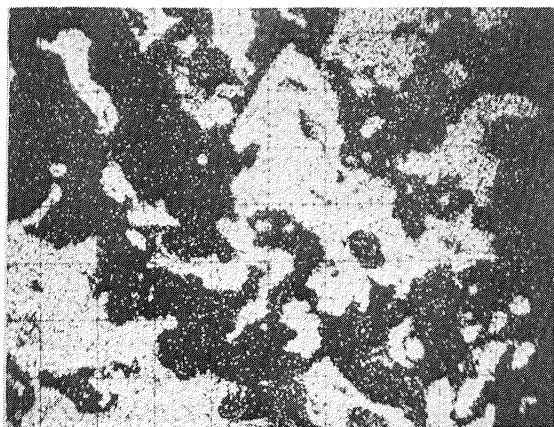




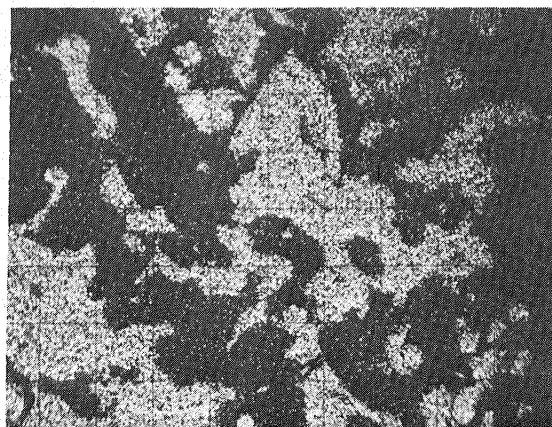
(a) Backscatter electron photograph.



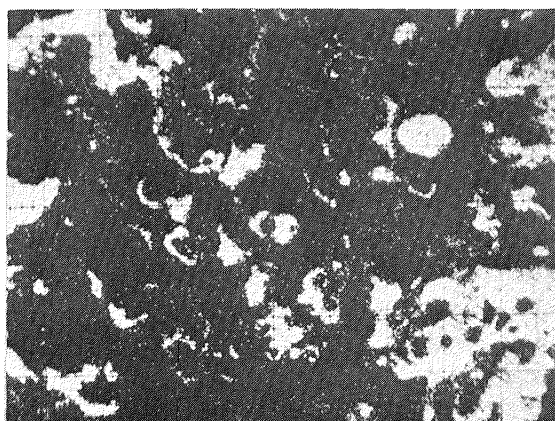
(b) Silver X-ray photograph.



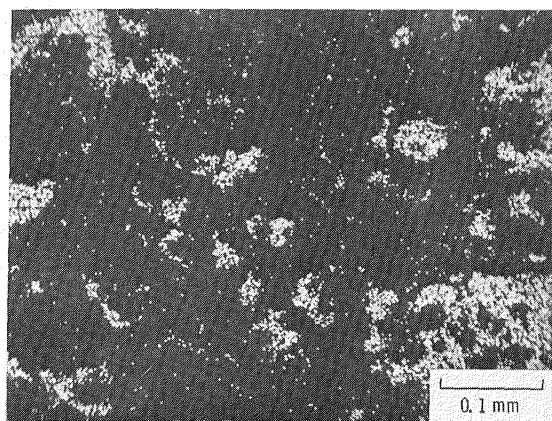
(c) Nickel X-ray photograph.



(d) Chromium X-ray photograph.



(e) Calcium X-ray photograph.



(f) Fluorine X-ray photograph.

Figure 5. - EMXA photographs showing elemental distribution in NASA LUBE PS106 coating 1 (with 1.25 wt %  $\text{AlPO}_4$ ).

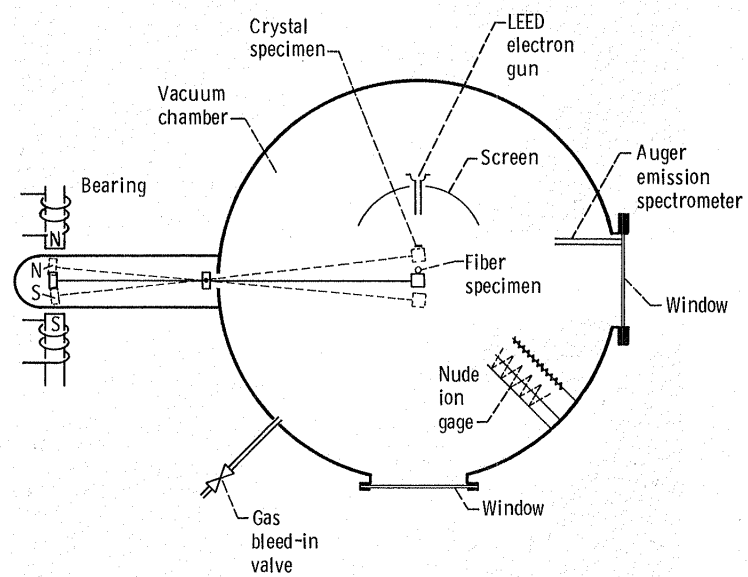
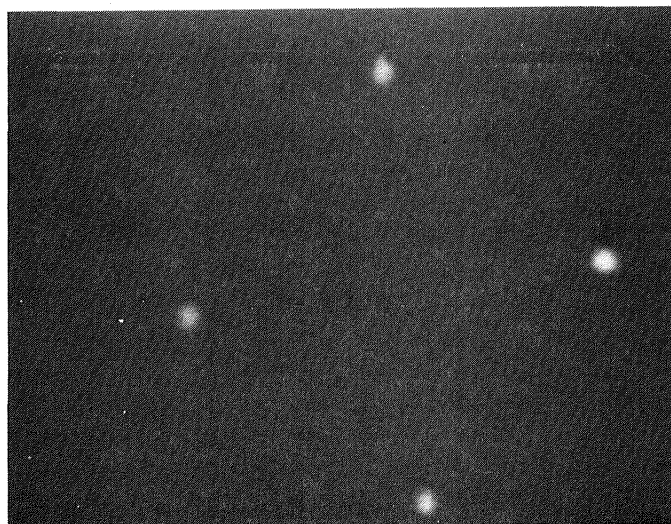


Figure 6. - LEED adhesion apparatus.





(a) Clean iron (001) surface.



(b) Methane - covered iron (001) surface.

Figure 7. - LEED patterns for an iron (001) surface in the clean state and with a monolayer of methane present.

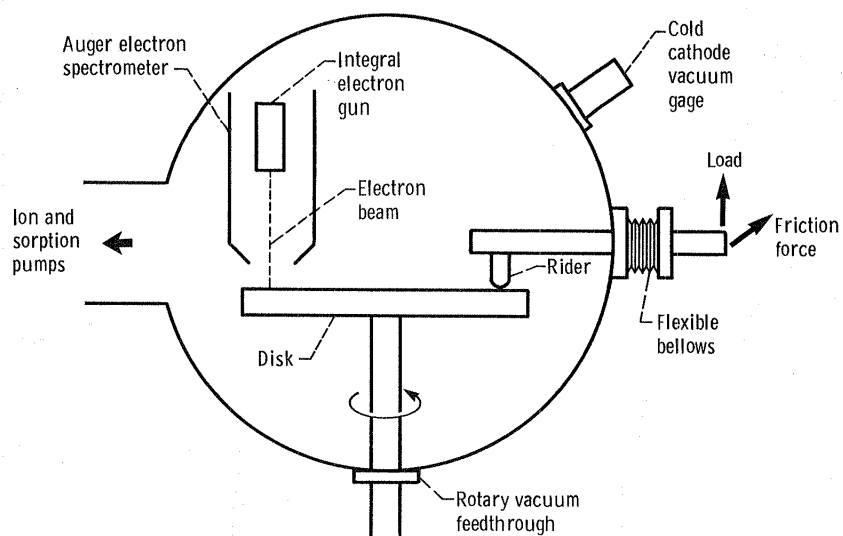


Figure 8. - Friction apparatus with auger electron spectrometer.

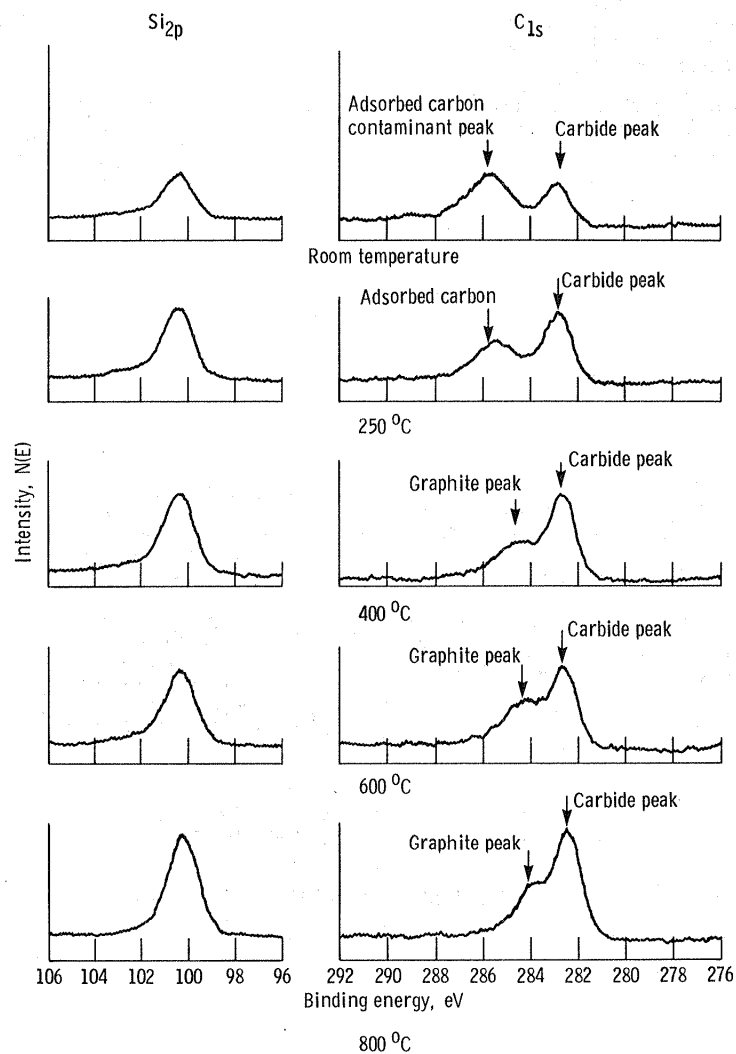


Figure 9. - Representative Si<sub>2p</sub> and C<sub>1s</sub> XPS peaks on SiC surface preheated to 800 °C.

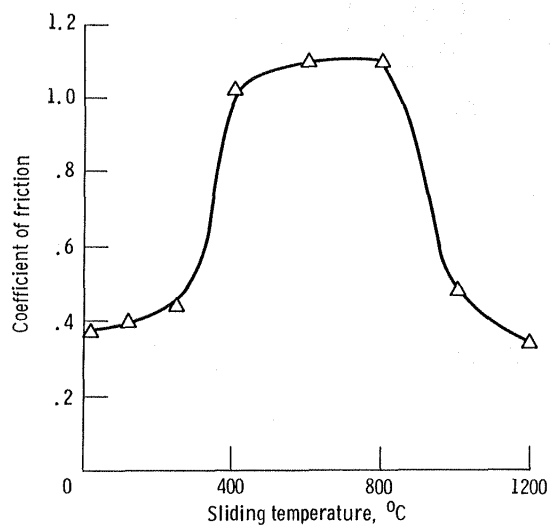


Figure 10. - Effect of temperature on friction for sintered polycrystalline SiC. Iron sliding against SiC; vacuum, 30 nPa; normal load, 0.1 to 0.2 N.

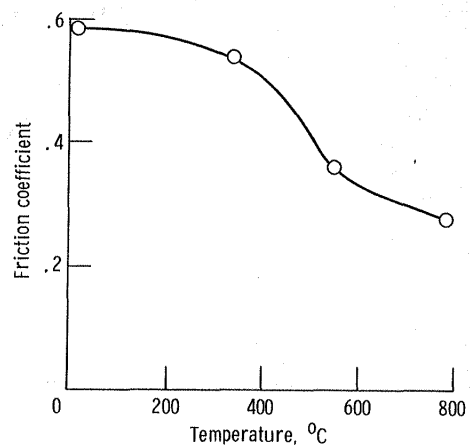


Figure 11. - Friction versus temperature for silicon carbide versus IN750. Rub shoe load, 67/N; disk speed, 0.18 m/sec; atmosphere, room air.

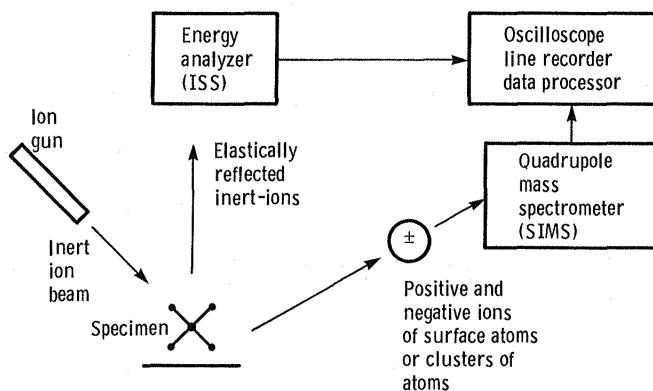


Figure 12. - Outline of the ISS/SIMS surface analyzing system.

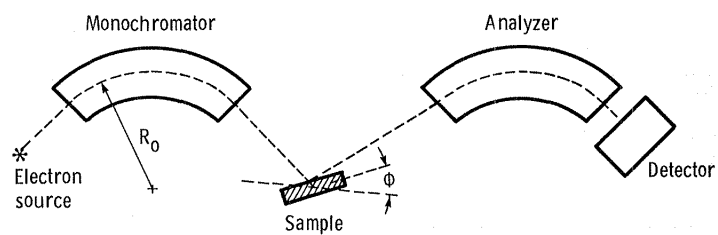


Figure 13. - Schematic diagram of electron energy loss experiment. Electrons from cathode pass through monochromator, strike sample, and energy spectrum of scattered electrons is probed by second monochromator.

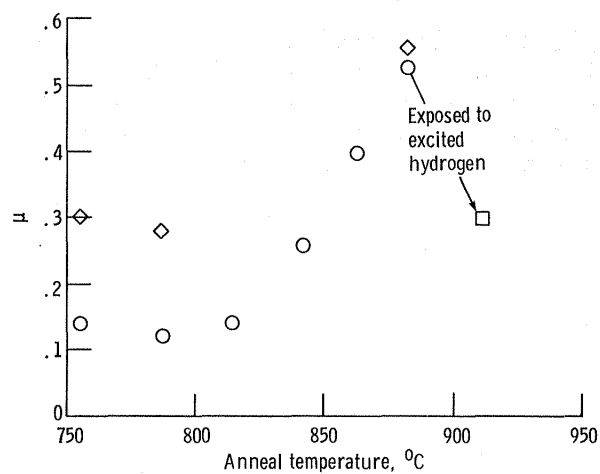


Figure 14. - Friction of diamond in vacuum.

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16. Abstract  Surface analytical tools are very beneficial in tribological studies of ceramics. Traditional methods of optical microscopy, XRD, XRF, and SEM should be combined with newer surface sensitive techniques especially AES and XPS. ISS and SIMS can also be useful in providing additional composition details. Tunneling microscopy and electron energy loss spectroscopy are less known techniques that may also prove useful.					
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